WI 220804.06

T 1218

BALLOT NO. 02 SARG

DRAFT NO. 01

DATE 6/1/2023

WORKING GROUP CHAIR To Be Determined

SUBJECT CATEGORY Optical Properties

RELATED METHODS See "Additional Information"

CAUTION:
This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Calibration of reflectance standards for hemispherical geometry

(Five-year review of Standard Practice T 1218 sp-18: Approval of T1218 Draft 1)

1. Scope

1.1 This standard practice describes the calibration of standards for hemispherical reflectance in relation to the theoretically perfect reflecting diffuser with an assigned value of unity.

1.2 The calibration of an instrument standard is made by means of a standard coated flat plate.

1.3 The absolute reflectance of the flat plate is determined with a spectrophotometer equipped with an integrating sphere to which has been added an auxiliary sphere.

1.4 The flat plate is coated at the same time, and in the same manner as, the interior of the auxiliary sphere, so that it is essentially a flat segment of the sphere's interior surface. The flux reflected from the interior of the auxiliary sphere through its port is compared with that reflected from the flat plate. The absolute or total diffuse reflectance of the flat plate is calculated by comparing the two reflected fluxes and the ratio of the port area to the wall area of the sphere.

2. Significance

The flat plate is assumed to have a perfectly diffusing surface, but it may possess structural characteristics that will influence a reflectance determination by the way in which the surfaces are illuminated and viewed. The spectrophotometer used for determining an absolute reflectance ideally should have the same optical geometry as the instrument for which the standard is calibrated. The absolute reflectance obtained using this auxiliary sphere method is for diffuse-diffuse (*d/d*) geometry. Most instruments employed for the measurement of reflectance in the paper industry have either 45/0 geometry or *d*/0 geometry. These differences in geometry between the auxiliary sphere system and the test instruments means that significant differences can result when different materials are employed as transfer standards. Translucency effects must also be considered. If the transfer materials used have similar characteristics with regard to scattering, absorption, and goniophotometric curves, then the differences relating to geometry will be fixed.

3. Apparatus

3.1 *Spectrophotometer*, capable of measuring hemispherical reflectance by means of an integrating sphere and having a photometric precision of 0.001.

3.2 *Auxiliary sphere,* of metal and made in two halves that can be taken apart easily for coating[1]. One half has an entrance port to admit light to the sphere. The edge of this port must be finely beveled. The other half has a removable flat plate so located that its center coincides with the center of the irradiating beam. The diameter of the entrance-port of the auxiliary sphere is larger than the diameter of the irradiating-beam and slightly smaller than the diameter of the outer-port in the instrument sphere (see Fig.1). The diameter of the auxiliary-sphere is such that its internal area is of the order of 100 times the area of its port. The interior surface of the sphere should be roughened, as by sand blasting, to facilitate adhesion of the coating.

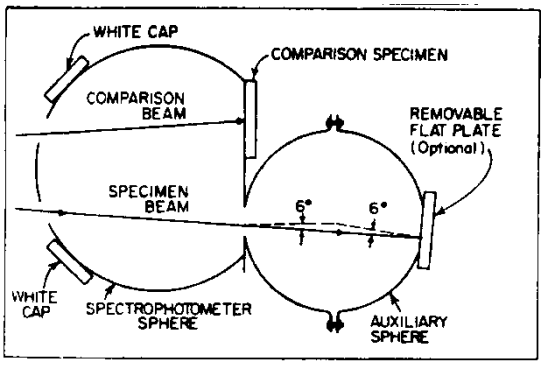

Fig. 1. Application of auxiliary sphere to spectrophotometer

[1] Alternatively, the sphere may be constructed without this removable plate, with a separate flat plat about 100 × 100 × 5 mm, provided to be coated along with the sphere.

3.3 *Jig for coating sphere*, with pressed powder, constructed of the following parts: (a) a semicircular plastic blade with a tapered edge, (b) an accurately centered bearing on which to rotate the blade to form a hemisphere; and (c) a guard ring of plastic to be mounted on the larger hemisphere flange to prevent chipping of the edge of the formed coating (see Fig. 2). The radii of the flange and semicircular blade are made 3 mm smaller than the larger sphere radius so as to produce a coating 3 mm thick.

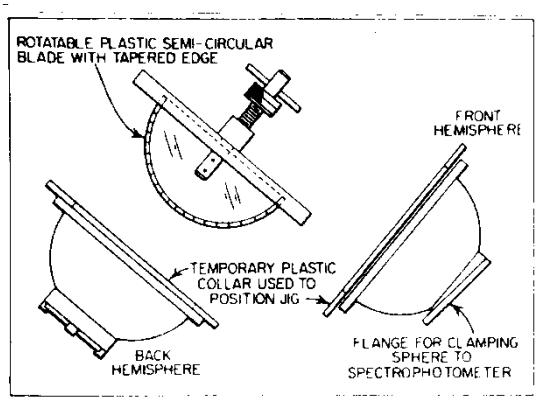

Fig. 2. Hemispheres and jig for scooping out pressed powder.

3.4 *Flat plates*. When the sphere is designed without a removable flat plate, two separate flat plates, about 100 by 100 by 5 mm, are provided for coating at the same time as the sphere. These are provided with a cylindrical recess having a diameter of 1.2 times that of the port of the instrument sphere. The depth of this recess is 1 mm for paint and 3 mm for pressed powder.

3.5 *Coating materials*. Suitable materials for coating spheres and plates are: (a) reagent grade barium sulfate powder, (b) reagent grade magnesium oxide powder and (c) spectralon or an equivalently lambertian and opaque material with equal or greater reflection characteristics.

3.6 *Instrument standard*, a durable standard such as pressed barium sulfate, porcelain enamel, or white opaque glass.

3.7 *Preparation of spheres and plates*. Coat the sphere uniformly and thick enough so that any variation of thickness does not cause a variation in its reflectance. If there is a removable plate, the sphere should be coated with the plate in place so that there will be no crack in the final finish or coating. When the sphere halves are joined, the crack between the two halves should be filled with the same coating material so that no crack is visible. Flat specimens must be made of the same material and in such a manner that their reflectance is identical with that of the coating of the sphere. (a) *Coating auxiliary sphere; pressed-powder procedure*. Cover the entrance port of the sphere with masking tape. Form the coating by partially filling each hemisphere with MgO or $BaSO_4$ powder, using a plastic blade to press the powder against the wall of each hemisphere. Attach the jig to each hemisphere and carefully scoop out the excess powder and invert it frequently to remove loose powder. Save the powder for the flat plate. Remove the jig and lightly smooth the coating with a camel's hair brush. Lay the hemisphere on a table with the entrance port up and remove the tape. Trim the excess powder from the edge of the port by running a fine steel wire around the edge at an angle of 45°. Bevel the coating near the port against the sphere with a cone-shaped plastic mandrel previously

pressed into the powder and then inserted through the port to provide a smooth, thin edge. (b) *Coating auxiliary sphere; paint procedure*. Make a sphere paint by dispersing reagent-grade MgO or $BaSO_4$ in high-purity alcohol or by following manufacturer's instructions. The consistency should be as heavy as a spray gun can handle and still provide a uniform coating. Apply numerous thin coats, allowing sufficient time for drying between the coats, until the thickness of coating is at least 1 mm. Smooth the final surface by lightly brushing with a camel's hair brush soon after drying. (On some materials, brushing with a camel's hair brush may not be necessary.)

NOTE 1: In the event that dispersion with alcohol may present a fire hazard, make a water dispersion using a detergent dispersing agent. In either event both the sphere and the reference should be coated in the same manner.

3.8 *Coating separate flat plates*. When the flat plate is not to be removed from the sphere, or when the sphere is not provided with a removable plate, coat two separate plates to provide a measure of reproducibility of the coating technique: (a) *Pressed powder*. Press any powder removed when coating the hemispheres into the recess in each plate. Remove the excess powder by scraping with a straightedge made of the same material as the blade used to scoop out hemispheres. (b) *Paint*. Paint the flat plate at the same time the hemispheres are painted by using enough coats of paint to fill the recess in the plate. Remove any paint from the face of the plate beyond the recess before applying a succeeding coat. Smooth the surface of the final coat as indicated for the hemispheres.

4. Procedure

4.1 Operate the spectrophotometer in accordance with the instructions of the manufacturer. If it has specular ports, place white caps over them.

4.2 Obtain spectral reflectance data Q_s, for the auxiliary sphere. Measure spectral reflectances at least every 10 nm, and preferably every 5 nm, over the range of 340 to 780 nm or at least over the wavelength range within which the standard will be used.

4.3 Remove the flat plate or plates from the auxiliary sphere and obtain spectral reflectance data, Q_F for each flat plate coated with the same material as the sphere.

4.4 When the spectral reflectance data for the two separate plates do not agree to within 0.002(0.2%), the method of preparation is deemed to be unsatisfactory and the coating technique should be improved and repeated. If the plate is part of the sphere, remove it carefully by disassembling the hemispheres and scoring the coating at the edge where the plate is tangent to the sphere to avoid destroying the coating on the plate.

4.5 Measure the diameter of the port in the sphere and the average inside diameter of the coated sphere.

4.6 Using the coated flat plate as a reference, determine the reflectance of the instrument standard.

5. Calculations

5.1 Calculate the factor *f*, the ratio of the area of the spherical surface occupied by the entrance port to the total area of the sphere including its port, as follows:

$$f = 0.5\left[1-\sqrt{1-(r/R)^2}\right]$$

where

r = radius of the port

R = radius of the sphere

5.2 Calculate the ratio of instrument reading Q_F for the flat plate to Q_s for the sphere at each wavelength.

5.3 Calculate the absolute reflectance of the flat plate, ρ_F at each wavelength as follows:

$$\rho_F = \frac{1 - f(Q_F / Q_S)}{(1 - f)}$$

5.4 Calculate the absolute reflectance ρ_R, of the instrument standard from Q_R instrument reading for the instrument standard and Q_F, as follows:

$$\rho_R = \frac{Q_R}{Q_F} - \rho_F$$

6. Report

Report the absolute reflectance of the instrument standard at convenient wavelength intervals (at least every 10 nm in the visible spectrum). Identify the auxiliary sphere coating and the spectrophotometer used. Compare the spectral reflectance data obtained for the instrument standard with those obtained in previous determinations.

7. Precision and accuracy

A precision statement is not applicable for this method; however, if the flat plate reflectance is not identical to that of the sphere coating, a substantial error will result when the absolute reflectance of an instrument standard is calculated from the reflectance of the flat plate. When the ratio of the sphere area to the port area is 100:1, a difference of 0.025 between the flat plate reflectance of the sphere coating affects the absolute determination of ρ_F by only 0.001, but the instrument standard calibrated relative to the flat plate will be in error by 0.026 because the plate is not representative of the sphere coating. Such difficulty is least likely to occur when the pressed powder technique is used for coating both the sphere and flat plate.

8. Keywords

Calibration, Standards, Reflectance, Geometry

9. Additional information

9.1 Effective date of issue: To Be Assigned

9.2 The best procedure for producing a sphere coating of high reflectance and good uniformity is with pressed powder and jig as described in this method. The jig provides an easy method for obtaining a coating with an accurately known diameter, an import prerequisite to determine absolute reflectance. Whatever method is used for coating the sphere, a flat plate with an identical coating is required.

9.3 With paint there is some question of whether the reflectance of the plate is identical with that of the sphere. The coating thickness may vary, and it may not be sufficiently thick at all points to produce the maximum reflectance of the material.

9.4 A related method is ASTM E-306.

9.5 This procedure was formerly T 679. It was withdrawn on December 3, 1984 and became TIP 0804-07. In 1998, it was returned to the test method set as a Standard Practice.

References

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4. Venable, W.J., Jr., Hsia, J.J., and Weidner, V.R., "Establishing a Scale of Directional-Hemisphere Reflectance Factors; I: The Van den Akker Method," *J. Res. Nat. Bureau of Standards* **82**(1):29 (1977).

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department.

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